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MICROWAVE-ASSISTED PYROLYSIS OF OIL PALM SHELL BIOMASS

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ABSTRACT

A new technique was developed for microwave pyrolysis in which inert nitrogen gas was supplied from the top of the reactor. It is established that by the use of this technique, the problem of bio-oil deposition onto the reactor walls was solved. The pyrolysis of oil palm shell (OPS) was conducted in a microwave oven using an activated carbon as microwave absorber. The temperature profiles, product yield, as well as the properties of the pyrolysis products were found to be effected by the amount of microwave absorber and the stirrer speed, which this fact can be used to control the product quality. It was found that increasing the amount of microwave absorber as well as the stirrer speed reduced the yield of bio-oil. An increase in microwave absorber percentage has the effect of increasing the amount of phenol present in the bio-oil with maximum of 84.76 area % phenol obtained at 75% microwave absorber at 100 rpm stirrer speed. The maximum bio-oil yield of 28 wt. % was obtained at 25% microwave absorber at 50 rpm stirrer speed. The maximum calorific value of the bio-char was found to be 29.5 MJ/kg obtained at 50% microwave absorber at 100 rpm stirrer speed. High calorific value of the biochar could be utilised as solid fuel in many heating applications. Furthermore, the GC-MS characterisation of the bio-oil shows that the constituents are mostly phenol and its derivatives which are valuable chemicals having a wide range of applications and the potential to be an alternative to their petroleum-derived counterparts.

Keywords : *Microwave, Fast pyrolysis, Oil palm shell (OPS), Bio-oil, Biomass*

1.0 INTRODUCTION

The unsustainable nature of fossil fuels, rapid increase in their cost, and environmental impact associated to their usage are some reasons that led to the increased interest in alternative sources of energy and/or chemical feedstock. Biomass is primarily a hydrocarbon material consisting mainly of carbon, hydrogen, oxygen, nitrogen, and sometimes traces of sulphur (Yaman, 2004). When used as a feedstock in a thermo chemical conversion process, biomass can be the only renewable energy source that can be transformed directly into solid, liquid, and/or gaseous fuels/chemicals (Bridgewater and Peacocke, 2000). Oil palm shell (OPS) is one of the most common biomass wastes in Malaysia generated through the oil palm milling process from various palm oil mills located in the country with an annual estimate of 2. 4 million tonnes of palm shell waste recorded (Abnisa et. al. 2011).

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Pyrolysis is a thermal decomposition of a material occurring in the absence of oxygen, it can be categorised as flash (favouring gas production), fast (favouring liquid production), or slow (for solid char production) by controlling the heating rate and the residence time for the process (Bridgewater, 2011). An alternative to the conventional heating commonly used during pyrolysis is the use of microwave radiation which is gaining many advantages in wastes management and other industrial heating applications (Appleton et. al, 2005). Among the major advantages recorded for microwave heating used in pyrolysis process over the conventional heating are faster and more uniform heating, direct utilization of a large-sized particles as feedstock, better production quality, and enhanced chemical reactivity (Yolanda et. al. 2011). Also the drying action through moisture removal of microwave (Wang et. al. 2008), can eliminate the need for separate moisture removing process before biomass pyrolysis when microwave is used for the whole process. Not all materials absorb microwaves. Because of this, microwave absorber like activated carbon is normally added to facilitate heating in microwave-assisted pyrolysis process.

Many literatures were found on the microwave-assisted pyrolysis of different wastes materials among which are sewage sludge (Wei Zuo et.al, 2011), scrap tyres (Ani and Syarizan, 2011), plastics (Zahid et. al. 2012), and waste printed circuit boards (Zhang. et. al., 2010). Literatures found on microwave-assisted pyrolysis of biomass include sawdust pellet (Shoujie Ren et. al. 2012), empty fruit bunch (Omar et. al. 2011), coffee hulls (Dominquez et. al, 2007), rice straw (Du Jin et. al and Huang et. al, 2010), wheat straw (Vitaly et. al, 2009), woods (Krzan and Ema Zagar, 2009, Robinson et. al, 2010), corn stalk bale (Zhao et.al, 2008), distillers dried grain (Hanwu Lei et. al, 2011), lignocellulosic biomass (Quan Bu et. al, 2011), corn Stover (Hanwu Lei et.al, 2009), and oil palm fibers (Ani and Salema 2011) all of which involves the use of microwave pyrolysis to convert these biomasses into useful fuels and/or chemicals.

The only literature found on the microwave induced pyrolysis of oil palm shell (Salema and Ani, 2011) looks at the effect of microwave absorber on temperature profile and bio oil yield. Also, the same parameters were investigated by the same authors, but an overhead stirrer (with constant speed) was used in this instance to facilitate the process (Salema and Ani, 2012). There is however no literature found on microwave-induced pyrolysis of OPS that investigate the effect of microwave absorber and stirrer speed on temperature profile, pyrolysis product yield, and the properties of the final products.

The objective of this work was to study the effect of microwave radiation on OPS with and without the use of char as a microwave absorber. Different microwave absorber percentage as well as stirrer speed was investigated as regards temperature profile, bio oil yield, and the characteristics of the pyrolysis products. Furthermore, one of the problems encountered in our lab previously was that of bio-oil deposition on the reactor walls which causes loss in bio-oil and a tedious clean-up process after each experiment. In this study, an attempt was made to solve this problem by developing a new technique for microwave pyrolysis through the supply of nitrogen gas from the top of the reactor and the pyrolysis vapours are channelled and collected down for condensation.

2.0 MATERIALS AND METHODS

2.1 MATERIALS

The Oil palm shell (OPS) biomass used in this experiment was acquired from an Oil palm mill, Felda, Kulai, located in Johor Bahru area of Johor in Malaysia. The oil palm shell was ground to smaller particles of 1.4 mm size to facilitate heating process. The moisture content of the OPS at the time it was acquired was found to be 8.4 wt%. The microwave absorber used was coconut-based activated carbon of 3 mm particle size obtained from Laju Group of Companies, Malaysia. The bigger particle size of the microwave absorber was essential to make sure that no carbon particles escapes together with vapours into the condenser and subsequently into pyrolysis oil through the distributor plate. Table 1 presents both proximate and ultimate analysis of the OPS biomass on dry basis.

Table 1 : Characteristics of Oil Palm Shell on dry basis

Proximate analysis (wt. %)		**Ultimate analysis (wt. %)**	
Moisture	8.5	C	55.35
Volatile	68.8	H	6.43
Fixed Carbon	20.3	N	0.37
Ash	2.3	O	38.01

2.2 Experimental set-up

The schematic diagram shown in Figure 1 represents the experimental set-up for the microwave-assisted pyrolysis system. A domestic microwave oven characterised by 800 W maximum power and 35 minutes maximum time selection was modified to accommodate the quartz glass reactor used to carry out the experiments. The reactor is 150 mm in length having 100 mm diameter. Two glass lids were used to cover the reactor by clipping them to it at both ends. The first one with tree openings arranged in a straight line was used to cover the top of the reactor and the other with one opening at its centre was used to cover the bottom of the reactor. In-between the bottom cover and the reactor, a wire mesh of 80 micron diameter was used to support the biomass mixture as well as allow the vapours generated to escape down into the condensing unit.

A Y-shaped glass connector was used in the central opening of the top cover of the reactor to allow the use of an overhead stirrer and nitrogen gas supply into the reactor, in the other two openings of the top cover, two K-type thermocouples wires were inserted into the reactor. The stirrer is a 7 mm wide double-bladed stainless steel of 250 mm length which can be adjusted until a suitable height needed for stirring action is achieved and is connected to a M590-502 speed controller motor (90 W) acquired from Tung electrical company ltd through which its speed can be controlled. The two thermocouples wires were connected to a Pico data acquisition system having an accuracy of $\pm 0.5°C$ which was also connected to a personal computer for constant monitoring of temperature during the experiment. The temperature T1 is the surface bed temperature of the biomass mixture, while T2 represents the inside bed temperature of the mixture. To ensure that no microwave leakages occur during the experiments, frequent monitoring of leakages was done using a MW leakage detector model TX90 acquired from Robin Professional Test Equipment, U.K.

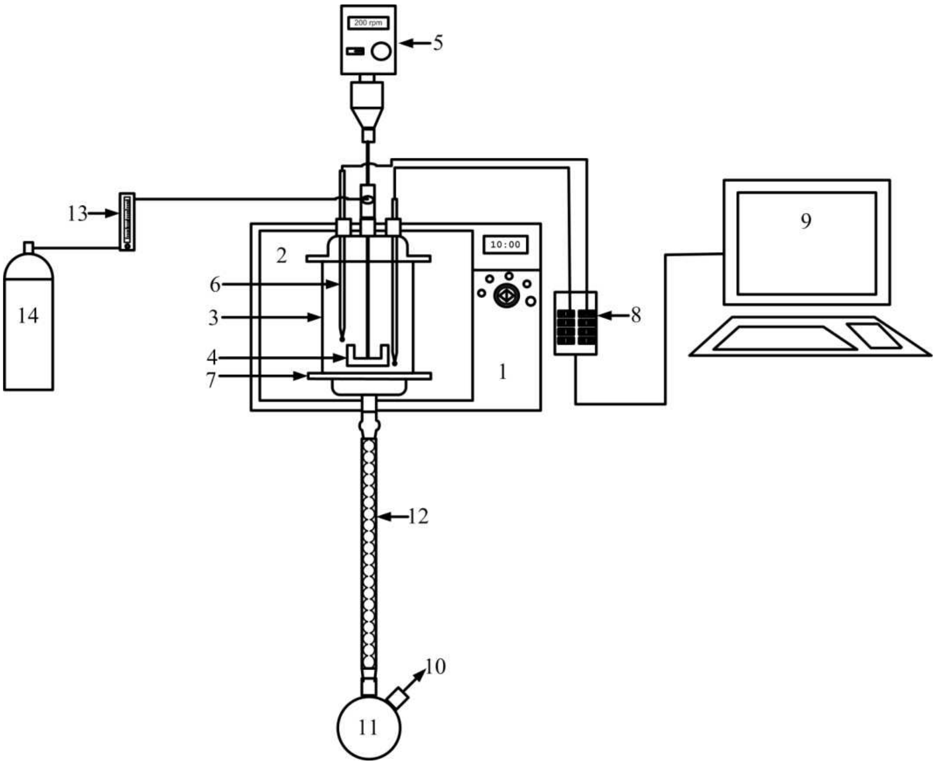

Figure 1: Experimental set-up, (1) Microwave control (2) Microwave cavity (3) Quartz glass reactor (4) Stirrer (5) Stirrer motor (6) K-type thermocouples wires (7) Wire mesh (8) Pico Data acquisition system (9) Personal computer (10) Uncondensed gases escape (11) Bio-oil collector (12) Condensers (13) Nitrogen gas flow meter (14) Nitrogen gas tank

2.3 METHODS

Before commencing each experiment, pure nitrogen gas was supplied at the rate of 7 LPM (litres/minute), this was to ensure that an inert environment was created in the reactor. This continued throughout the reaction to make sure that the environment created was maintained as well as sweep the pyrolysis vapours out of the reactor for condensation. The nitrogen flow rate was not reduced during the experiment to make sure that the vapours which will naturally tend to go up are suppressed down and immediately channelled into the condenser, this help solve the problem of bio-oil condensation on the reactor walls as well as the connections to condensers. All of the experiments were conducted using fixed a microwave power of 450W which our previous research established that it is adequate for pyrolysis of OPS and going beyond that does not help the pyrolysis final product or the economy of the system (Salema and Ani 2012). With the weight of the OPS biomass kept constant at 150g throughout the experiment, the percentage of the microwave absorber used was varied at 25%, 50%, and 75% of the weight of the biomass to determine the effect of microwave absorber on the system. Also, the stirrer speed was varied at 50rpm, 100rpm, and 150rpm to investigate the effect of stirrer speed. An iced-cooled water of around 10℃ in temperature was used to cool the condensers used for collecting vapours and subsequently bio-oil. After each experiment the weight of the bio-char as well as that of bio-oil was determined. With the weight of the biomass kept constant the yield of bio-oil as well as that of bio-char was determine as weight percentages. The difference between the two was used to calculate the weight percentage of uncondensed gases produced during the experiments**.**

2.3.1 GC-MS Characterisation of the bio-oil

An Agilent Technologies 6890 GC–MS device with HP-5MS capillary column was used in characterising the chemical content of the bio-oil. The solvent used was acetonitrile and helium gas was used as the carrier gas at a flow rate of 2ml/min. At the rate of 10 ◦C/min, the temperature of the oven was raised from 80◦C to 200◦C, and then at a rate of 5 ◦C/min to the final temperature of

300◦C which was held constant for 10 min. The injector temperature also was held constant at 250◦C. The identification of the chemicals contained in the oil was achieved by comparing the mass spectra generated in the MS with the ones available in the data library of the National Institute of Standards and Technology (NIST).

2.3.2 Calorific value of the bio-char

The gross calorific value of the bio-char was determined by the use of an IKA C2000 Bomb Calorimeter having an input power of 1.8 kW and 40000 J working range. Operating pressure of oxygen was 30 bar and the measurement procedure used was Isoperibolic 25°C that takes about 22 mins.

3.0 Results and Discussion

3.1 Temperature profiles

The pyrolysis temperature profiles obtained are shown in Figures 2 (a-f), T1 and T2 are the temperatures of the surface bed and inside bed of the biomass mixture respectively. The effect of microwave absorber on the temperature profile can be seen in Figures 2a, 2b, and 2c representing microwave absorber percentage of 25%, 50%, and 75% of the biomass weight respectively. It is evident from the figures that increase in microwave absorber has an effect of decreasing the pyrolysis temperature. This is in agreement with the result obtained by Salema and Ani (2012). It can be attributed to the fact that increasing the amount of the microwave absorber while keeping the power constant causes more molecules to compete for the same amount of radiation and consequently the overall temperature decreases. Another interesting observation is that of the increase in the surface bed temperature as the microwave absorber increases. At 25% microwave absorber, the surface bed temperature represented by the red line can be seen well below the inner bed temperature represented by the blue line. This however changes at 50% and 75% microwave absorber where it becomes even slightly higher than the inner bed temperature. The explanation of this was not found in the literatures but microwaves are known for their localised heating (Yolander et. al 2012) and increasing the receptor percentage in the mixture can increase this effect.

The stirrer speed has quite the opposite effect compared to the microwave absorber percentage as in this case increase in stirrer speed increases the pyrolysis temperature. Figures 2d, 2e, and 2f show the temperature profile obtained for 50 rpm, 100 rpm, and 150 rpm stirrer speed respectively. The maximum temperature attained during pyrolysis for the stirrer speed of 50 rpm, 100 rpm, and 150 rpm is 525°C, 606°C, and 650°C respectively for the same amount of microwave absorber (25%) used. Although the effect of increasing the stirrer speed on the temperature profile was not found in the literature, the use of the stirrer in pyrolysis process was found to produce higher pyrolysis temperature when compared with other studies conducted without it (Salema and Ani 2012).

The desired pyrolysis temperature of 500°C was obtained at 25% microwave absorber and 50 rpm stirrer speed (Figure 2a). The temperature of 500°C is considered suitable for fast pyrolysis since below this value the pyrolysis will not be rigorous enough and some of the biomass may still remain in the bio-char after the process. Going higher than this value have an effect of encouraging secondary reactions and hence more gaseous formation in the pyrolysis products (Rafael Luque et. al. 2012).

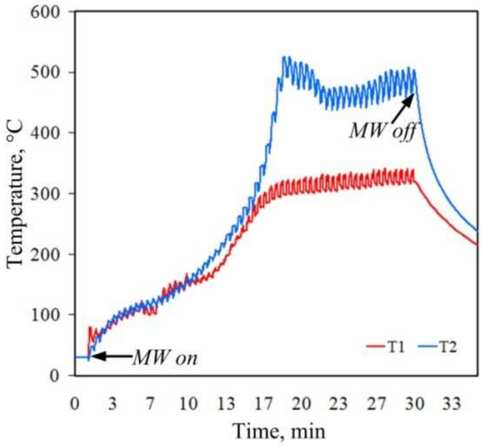

Figure 2a 25% MW absorber

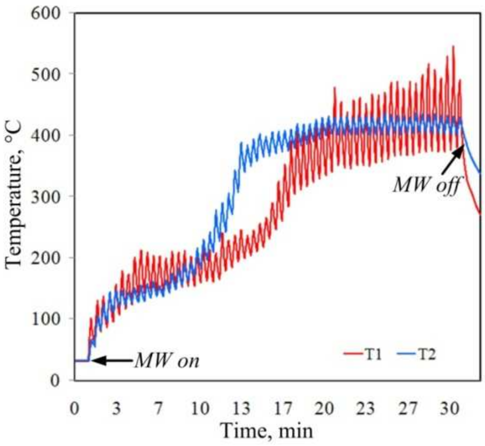

Figure 2b 50% MW absorber

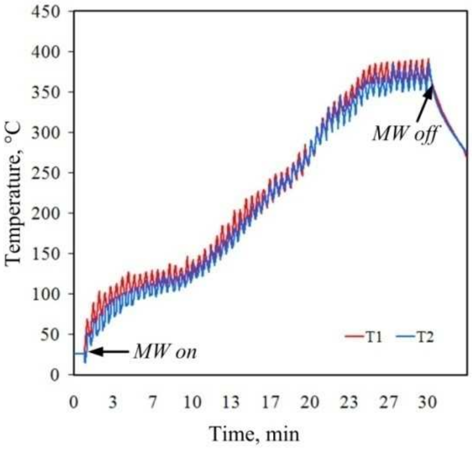

Figure 2c 75% MW absorber

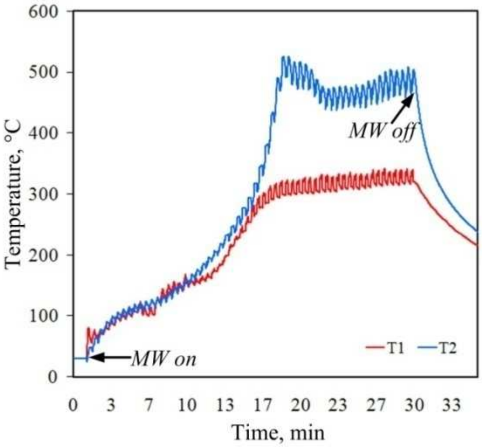

Figure 2d 50 rpm stirrer speed

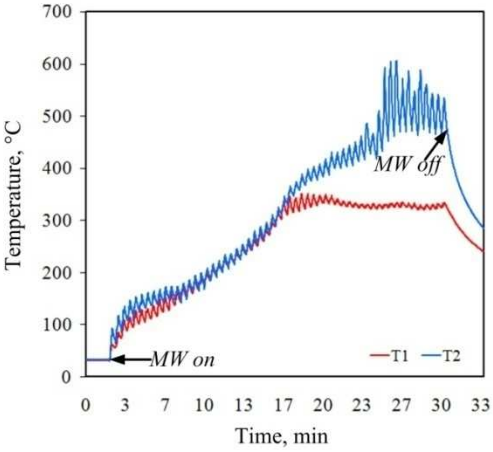

Figure 2e 100 rpm stirrer speed

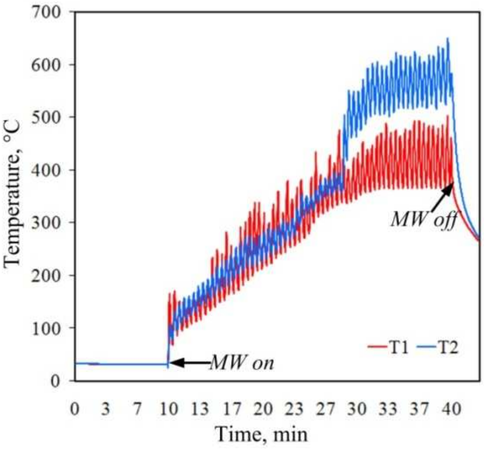

Figure 2f 150 rpm stirrer speed

3.2 Pyrolysis products yield

The effects of microwave absorber percentage and stirrer speed on pyrolysis product yield are shown in Figure 3a and 3b respectively. From Figure 3a, we see that bio-oil yield decreases with increase in microwave absorber percentage. The highest bio-oil yield of 28 wt.% was obtained at 25% microwave absorber. This can be explained if consideration is given to the maximum pyrolysis temperatures attained at the different microwave absorber; the temperature decreases with increase in microwave absorber with the desired pyrolysis temperature of 500°C obtained at 25% MW absorber. Also the figure shows decrease in bio-char percentage accompanied by increase in gaseous products as the microwave absorber was increased, although the bio-char percentage at 50% and 75% microwave absorber are almost the same.

The effect of stirrer speed on the product yield depicted in figure 3b shows the same pattern as that of microwave absorber percentage in that increase in the stirrer speed is accompanied by the decrease in bio-oil yield. It is important to mention again here that increasing the stirrer speed from 50 rpm, 100 rpm, to 150 rpm leads to the increase in temperature of the system. This increase in temperature evidently has effect on the bio-oil yield decrease as very high temperatures always encourage more gaseous product formation instead of bio-oil. This explanation agrees with the one reported by Quan Bu et. al (2011) although direct comparison could not be used due to differences in experimental conditions and the feedstock used.

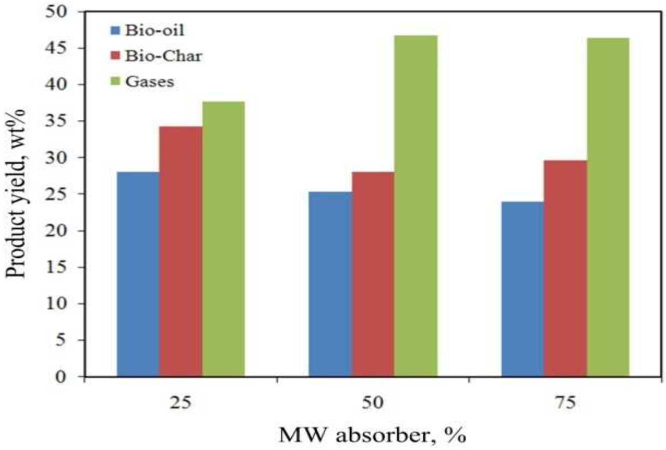

Figure 3a Effect of MW absorber on yield

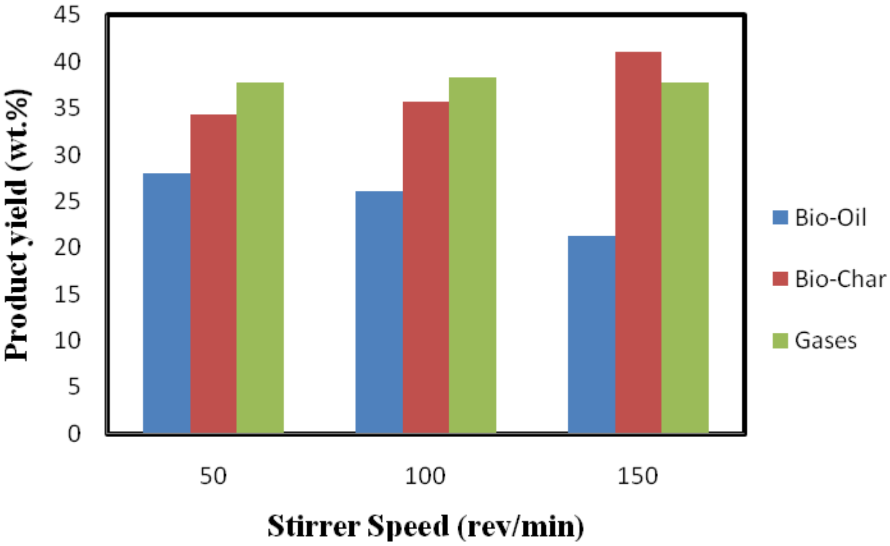

Figure 3b Effect of stirrer speed on yield

3.3 Calorific value of the Bio-char

The effect of microwave absorber as well as the stirrer speed on the calorific value of the bio-char is shown in Figure 4. From the figure we see that generally, increasing the stirrer speed decreases the calorific value of the bio-char. The percentage of the microwave absorber however does not display linear characteristics as regards its effect on the calorific value of the bio-char, but rather the maximum calorific value of 29.5 MJ/kg for the bio-char was obtained at 50% microwave absorber percentage. This however drops with increase or decrease in the percentage of microwave absorber away from 50%. Therefore, the maximum calorific value for the bio-char was obtained at 50% microwave absorber and 50 rpm. Although no literature was found that will throw more light as to what is the explanation behind this, it could be predicted that higher temperatures associated with higher stirrer speeds might have resulted into localised heating of the biomass and consequently less rigorous pyrolysis that can definitely lower the bio-char calorific value or perhaps the higher temperatures has an effect of initiating burning process of the bio-char.

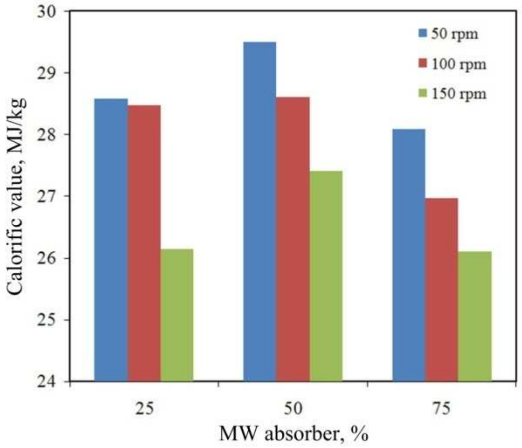

Figure 4 Effect of stirrer speed and MW absorber on calorific value of the bio-char

3.4 GC-MS Characterisation of the bio-oil

The chemical components identified in the bio-oil were basically phenol and phenolics (Tables 2 and 3). This agrees with some researches that uses lignocellulosic materials as their feedstock (Quan Bu et. al, 2011), (Hanwu Lei et. al, 2009 and 2011), and (Shoujie Ren et. al, 2012) even though some of these literatures also reported the presence of some aromatic and aliphatic compounds. From the Table 2 and 3, the phenol content in the bio-oil is higher compared to any of its derivative found in it. This is ascertained from the semi-quantitative analysis conducted in which each of the relative percentage of area formed by the constituent's chromatographic peaks was compared. The effect of MW absorber percentage on the chemical constituent's distribution in the bio-oil is shown in Table 2; increase in MW absorber percentage increases the amount of phenol in the bio-oil with the maximum of 84.76 area % obtained at 75% absorber percentage. Therefore the quantitative effect of MW absorber percentage for the bio-oil (in which its increase decreases the bio-oil yield) is being reversed qualitatively here through the increase in phenol content in the bio-oil whenever the percentage of MW absorber was increased**.**

The effect of stirrer speed on the chemical composition of the bio-oil is presented in Table 3. From the table we see that contrary to the linear effect displayed by the effect of MW absorber, the difference particularly in phenol content of the bio-oil as a result of stirrer speed is not very wide. The maximum value of 45.53 area % was obtained at 100 rpm but at 50 rpm and 150 rpm, traces of some aromatic compounds were found in the bio-oil. Also it is evident that the stirrer speed affects not only the temperature profile, products yield, and the calorific value of the bio-char but also the chemical distribution in the bio-oil. It is also important to mention here that even the lowest amount of phenol of 37.09 area % obtained in this research is higher compared to other researches using different feedstock and/or pyrolysis technology like that of Faisal Abnisa et. al. (2010), Kawser and Ani (2000), and Kin et. al. (2010). This shows that OPS can be a very good source of phenol and phenolics.

Table 2 : The chemical composition of the bio-oil at 100 rpm with different MW absorber amount

Chemical compounds	**Peak area %**		
	MW absorber, %		
	25	**50**	**75**
Phenol	44.1	45.5	84.7
2-methyl-phenol (o-cresol)	0.4	2.5	-
4-methyl-phenol (p-cresol)	-	1.5	-
2-methoxy-phenol	7.6	8.7	6.7
2-methoxy-4-methyl-phenol	5.2	6.6	2.1
4-ethyl-2-methoxy-phenol	-	6.7	-
2,6-dimethoxy-phenol	8.7	9.8	-
2-methoxy-4-vinyl phenol	1.7	-	-
2-methoxy-4-(1-propenyl)-phenol	2.3	-	-
2,6-methoxy-4-(2-propenyl)-phenol	2.4	-	-
Benzene	-	-	-
1,4-Benzendiol	-	-	-

(-) Not detected

Table 3 : The chemical composition of the bio-oil with different stirrer speeds

Chemical compounds	**Peak area %**		
	Stirrer speed, rpm		
	50	**100**	**150**
Phenol	38.9	45.5	37.1
2-methyl-phenol (o-cresol)	-	2.5	1.9
4-methyl-phenol (p-cresol)	-	1.5	1.9
2-methoxy-phenol (Guaiacol)	7.9	8.7	7.8
2-methoxy-4-methyl-phenol	5.9	6.5	5.8
4-ethyl-2-methoxy-phenol	-	6.7	6.2
2,6-dimethoxy-phenol (Syringol)	8.9	9.8	8.4
2-methoxy-4-vinyl phenol	1.3	-	1.0
2-methoxy-4-(1-propenyl)-phenol	2.2	-	-
2,6-methoxy-4-(2-propenyl)-phenol	2.0	-	1.4
Benzene	5.6	-	-
1,4-Benzendiol	-	-	1.5

(-) Not detected

4.0 Conclusion

Oil palm shell (OPS) biomass was pyrolysed using a new MW pyrolysis technique in which nitrogen gas was supplied from the top of the reactor and the pyrolysis vapour flow downward and bio-oil are collected at the bottom of the condensation system. It was established that using this set-up, the problem of bio-oil deposition on the reactor walls during pyrolysis was solved. From the results obtained, it is evident that the amount of Microwave absorber and the stirrer speed have an effect on temperature profile, product yield, as well as the properties of the bio-char and bio-oil obtained. The results show that increase in both stirrer speed and microwave absorber percentage decreases the bio-oil yield. This fact can be used to control pyrolysis processes without the use of any feedback control system. For this experiment, the desired pyrolysis temperature of 500°C was achieved at 25% MW absorber and 50 rpm stirrer speed. The highest calorific value of the bio-char was determined to be 29.5 MJ/kg and it could be used as solid fuel. Furthermore, the GC-MS analysis reveals that the content of the bio-oil are phenol and phenolics, very useful compounds having wide range of applications with the potential of being an alternative to their petroleum-based counterparts.

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